Supplementary Material

The R^2D^3 approach to fast quantitative NMR: maintaining accuracy and reducing experimental time

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Section 1: SPINit® pulse sequence and process

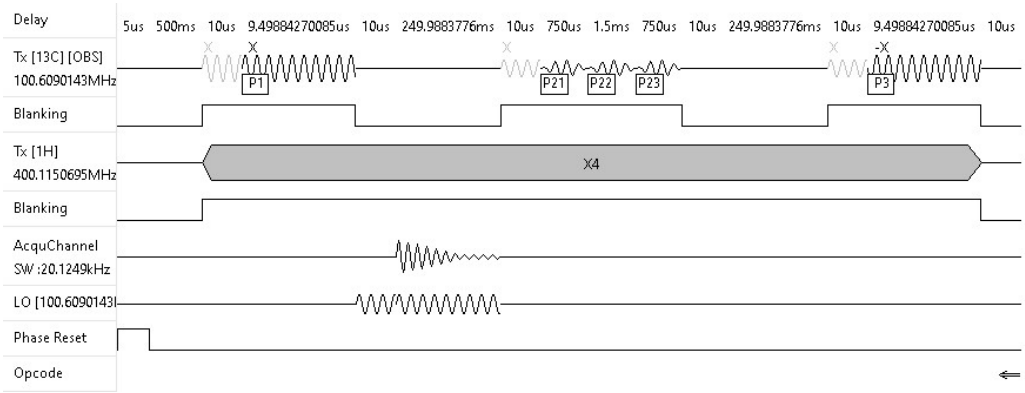


Figure S1: Graphical description of the sequence used in the programming interface. Since the sequence is programmed using only the graphical interface of SPINit®, it is not possible to provide a micro-program. However, jar files can be sent upon authors requests.

Transpose
Transpose the dataset such that the dimensions are cycle-shifted by one.
- Transpose order: Direct

Exponential
Apply a decreasing exponential apodization.
- Line broadening: 4000.0

Zero-filling
Increase the size of the rows and fill with zeros.
- Reconstruction size: 64
- Symmetric: false

Fourier transform for indirect dimension
Compute Fourier transforms adapted to indirect dimension on rows.
- Modulation: NONE

Transpose
Transpose the dataset such that the dimensions are cycle-shifted by one.
- Transpose order: Indirect

Projection
Projection
- Dimension: 2D
- Start: 27
- End: 35
- Operation: mean

Exponential
Apply a decreasing exponential apodization.
- Line broadening: 2.0

Forward linear prediction
Predict end points of a FID using a SVD method with a discrete regularization.
- Number of previous points: 400
- Reconstruction size: 20092

Constant baseline correction
Correct the baselines affected by a constant offset.
- Region without signal: End

Zero-filling
Increase the size of the rows and fill with zeros.
- Reconstruction size: 131072
- Symmetric: false

Fast Fourier Transform
Compute the Fourier Transform on rows using Tukey's algorithm.
- Shift: true
- Reverse: true
- Phase correction: false

Automatic phase correction
Correct automatically the phase of rows using a region-based method.
- independent rows: true

Figure S2: SPINit® screenshot of the process list for the R^2D^3 experiments, with an exponential apodization in the τ_r dimension.

Section S2: Simulations

All simulations were performed with PTC Mathcad Prime 9.0.0.0®

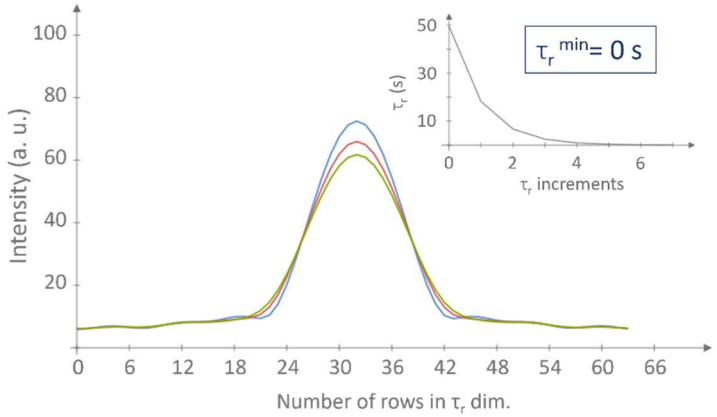


Figure S3: Simulation of the signal evolution in the τ_r dimension according to the number of rows for different T_1 (2 s blue, 3.5 s red and 5 s green). The τ_r function was an exponential with 8 values ending at $\tau_{r\,min}$ = 0 s, zero-filled to a final 64 rows in the τ_r dim. It was assumed that 85% of the magnetization was recovered after the DEFT block. No nOe was used.

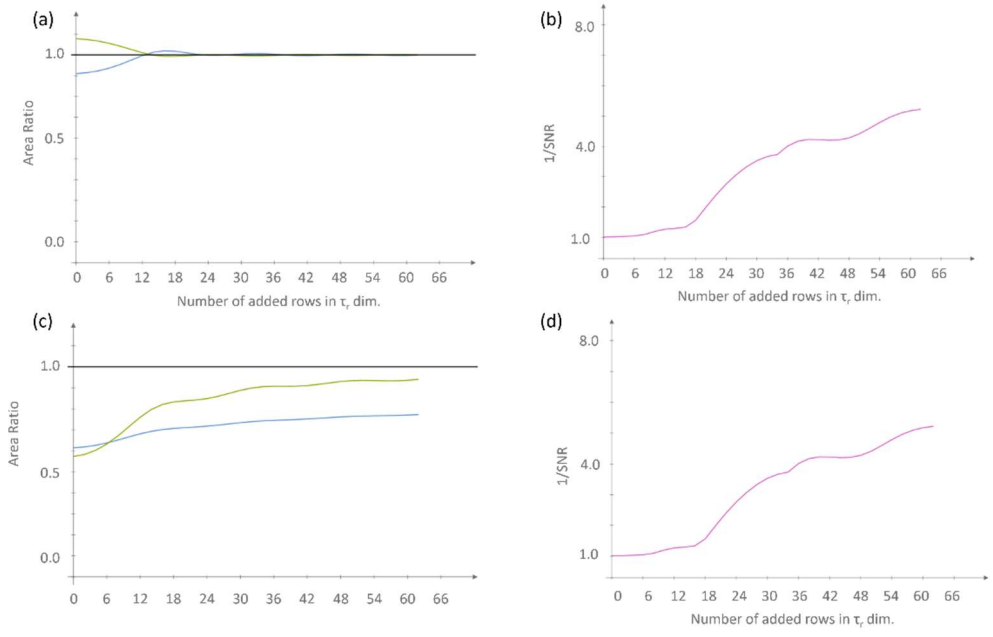


Figure S4: Simulation of the area ratio and the precision (1/SNR) depending on the number added rows in the τ_r dimension. In all cases, the τ_r function was an exponential with 8 values, zero-filled to a final 64 rows in the τ_r dimension. It was assumed that 85% of the magnetization was recovered after the DEFT block. Signals have the following characteristics: the first one has a T_1 of 3.5 s and η=1 for nOe, the second one has a T_1 of 5.0 s and η=0.5, the third one has a T_1 of 2 s and η=1. The green line is the area ratio of the first signal over the second and the blue line is the area ratio of the first signal over the third one. At first, no nOe was used and τ_r^{max} was $10 \cdot T_1^{max}$ (a and b); then nOe was added and τ_r^{max} was reduced to only $1 \cdot T_1^{max}$ (c and d).

Useful functions

Fourier Transfom (1D and 2D)

$$tf(v) := \left\| \begin{array}{l} u \leftarrow \mathrm{cfft}(v) \\ lon \leftarrow \mathrm{rows}(v) \\ \text{for } m \in 0..\frac{lon}{2}-1 \\ \quad \left\| \begin{array}{l} uu_{m} \leftarrow u_{m+\frac{lon}{2}} \\ uu_{m+\frac{lon}{2}} \leftarrow u_{m} \end{array} \right. \\ uu \end{array} \right.$$

$$mtf(w) := \left\| \begin{array}{l} lo \leftarrow \mathrm{cols}(w) \\ \text{for } l \in 0..lo-1 \\ \quad \left\| \begin{array}{l} wt \leftarrow w^{\langle l \rangle} \\ wtf \leftarrow tf(wt) \\ W^{\langle l \rangle} \leftarrow wtf \end{array} \right. \\ W \end{array} \right.$$

$$tf2D(v) := \left\| \begin{array}{l} u \leftarrow \mathrm{cfft}(v) \\ nbr \leftarrow \mathrm{rows}(u) \\ nbc \leftarrow \mathrm{cols}(u) \\ \text{for } m \in 0..\frac{nbr}{2}-1 \\ \quad \left\| \begin{array}{l} \text{for } n \in 0..\frac{nbc}{2}-1 \\ \quad \left\| \begin{array}{l} uu_{m,n} \leftarrow u_{m+\frac{nbr}{2},n+\frac{nbc}{2}} \\ uu_{m+\frac{nbr}{2},n+\frac{nbc}{2}} \leftarrow u_{m,n} \\ uu_{m+\frac{nbr}{2},n} \leftarrow u_{m,n+\frac{nbc}{2}} \\ uu_{m,n+\frac{nbc}{2}} \leftarrow u_{m+\frac{nbr}{2},n} \end{array} \right. \end{array} \right. \\ uu \end{array} \right.$$

Threshold

$$threshold(w,a) := \left\| \begin{array}{l} lo \leftarrow \mathrm{rows}(w) \\ \text{for } l \in 0..lo-1 \\ \quad \left\| \begin{array}{l} \text{if } w_l > a \\ \quad \left\| W_l \leftarrow w_l \right. \\ \text{else} \\ \quad \left\| W_l \leftarrow 0 \right. \end{array} \right. \\ W \end{array} \right.$$

Relaxation

$$St(a,b,c,d) := \left(1+d\cdot e^{\frac{-a}{b}}\right)\cdot\frac{\left(1-e^{\frac{-a}{b}}\right)}{1-c\cdot e^{\frac{-a}{b}}}$$

Noise generator

$$B(a,b) := \left\| \begin{array}{l} \text{for } l \in 0..b-1 \\ \quad \left\| W^{\langle l \rangle} \leftarrow (\mathrm{whiten}(a)+1\mathrm{i}\cdot\mathrm{whiten}(a)) \right. \\ W \end{array} \right.$$

Exponential

$$exp(x,a) := \exp(-a\cdot x)$$

Initial parameters

Full Width at Half Maximum

Relative intensity | | nOe (η)

$$A := \begin{bmatrix} 100 & -35 & 0.5 & 2 & -1 \\ 100 & 10 & 0.5 & 3.5 & -1 \\ 100 & 40 & 0.5 & 5 & -0.5 \end{bmatrix}$$

"Frequency shift" | T1 (s)

Reference molar fractions: $fref := \begin{bmatrix} 1 \\ 1 \\ 1 \end{bmatrix}$

τr max and min (s): $\tau\_r\_max := 45$ $\tau\_r\_min := 0.5$

Number of acquired points in both dimensions: $TD2 := 1024$ $TD1 := 8$

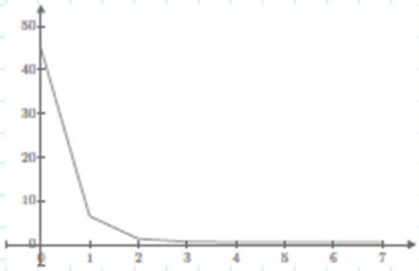
Percentage of recovered magnetization: $\lambda := 0.85$

Index: $i := 0..TD2 - 1$ $j := 0..TD1 - 1$ $N := \text{rows}(A)$ $n := 0..N - 1$

Time constant of the exponential: $b := 2$

τr function: $\tau\_r_j := (\tau\_r\_max - \tau\_r\_min) \cdot exp(j \cdot (b), 1) + \tau\_r\_min$

τr (in s) vs the row number (j)



FID generation parameters

$\Delta F2 := 100$ $\Delta t2 := \frac{1}{\Delta F2}$ $t2_i := i \cdot \Delta t2$

Noise generation

$MB := 0.15$ $nse := B(TD2, TD1)$ $Noise\_2D := 2 \cdot MB \cdot nse$

FID generation

$$FI_{i,j} := \left(\sum_{n=0}^{N-1} A_{n,0} \cdot e^{-1i \cdot \left(2 \cdot \pi \cdot A_{n,1} \cdot t2_i\right)} \cdot e^{-\pi \cdot t2_i \cdot A_{n,2}} \cdot St\left(\tau\_r_j, A_{n,3}, \lambda, A_{n,4}\right) \right) + Noise\_2D_{i,j}$$

FID processing

Processing parameters

Time constant of the exponential in the τ_r dimension: $pexp := 4$ $h_j := exp(j, pexp)$

Number of points in both dimension, after processing: $SI2 := 4 \cdot TD2$ $SI1 := 64$

Increments used for processing: $k := 0 .. SI2 - 1$ $l := 0 .. SI1 - 1$

Apodization

Exponential in the τ_r dimension: $FID_{i,j} := FI_{i,j} \cdot h_j$

Fourier transform

$S_{k,l} := 0$ $S_{i,j} := FID_{i,j}$

$Spec := tf2D(S)$ $SP_{k,l} := \sqrt{\mathrm{Re}\left(Spec_{k,l}\right)^2 + \mathrm{Im}\left(Spec_{k,l}\right)^2}$ $Sint := mtf(S)$

Profiles extraction

Increments: $p := 0 .. \left(\frac{SI1}{2} - 1\right)$ $o := 0 .. 5$ $ni := 0$ $nf := SI2 - 1$

Noise level measurment: $noises_o := \left(\mathrm{Re}(Sint)^{\langle 0 \rangle}\right)_{o+2}$ $se := 2 \cdot \max(noises)$

Peak picking: $spectrum := threshold\left(\mathrm{Re}(Sint)^{\langle 0 \rangle}, se\right)$ $D_{k,0} := k$ $D_{k,1} := spectrum_k$

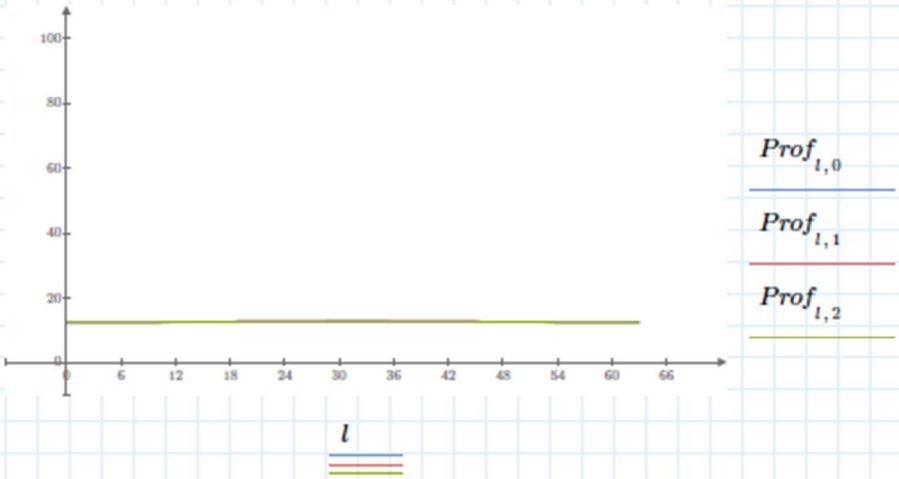
$Delta := \mathrm{localmax}(D)$ $\delta := Delta^{\langle 0 \rangle}$

Profiles

$$Proj_{k,p} := \sum_{l=\frac{SI1}{2}-p}^{\frac{SI1}{2}+p} Spec_{k,l} \qquad Base_p := \frac{\sum_{o=2}^{13} Proj_{o,p}}{12}$$

$$Projc_{k,p} := Proj_{k,p} - Base_p \qquad Prof_{l,n} := SP_{\delta_n,l}$$

Intensity profiles (a. u.) in the τ_r dimension vs the row number, after apodization (l)



Projection

$$f_{p,n} := \frac{N \cdot \mathrm{Re}\left(Projc_{\delta_n,p}\right)}{\sum_{n=0}^{N-1} \mathrm{Re}\left(Projc_{\delta_n,p}\right)}$$

$$\Delta f_{p,n} := 1000 \cdot \frac{f_{p,n} - fref_n}{fref_n}$$

Area ratio calculation

$$R_{p,n} := \frac{\mathrm{Re}\left(Projc_{\delta_i,p}\right)}{\mathrm{Re}\left(Projc_{\delta_n,p}\right)}$$

65

66

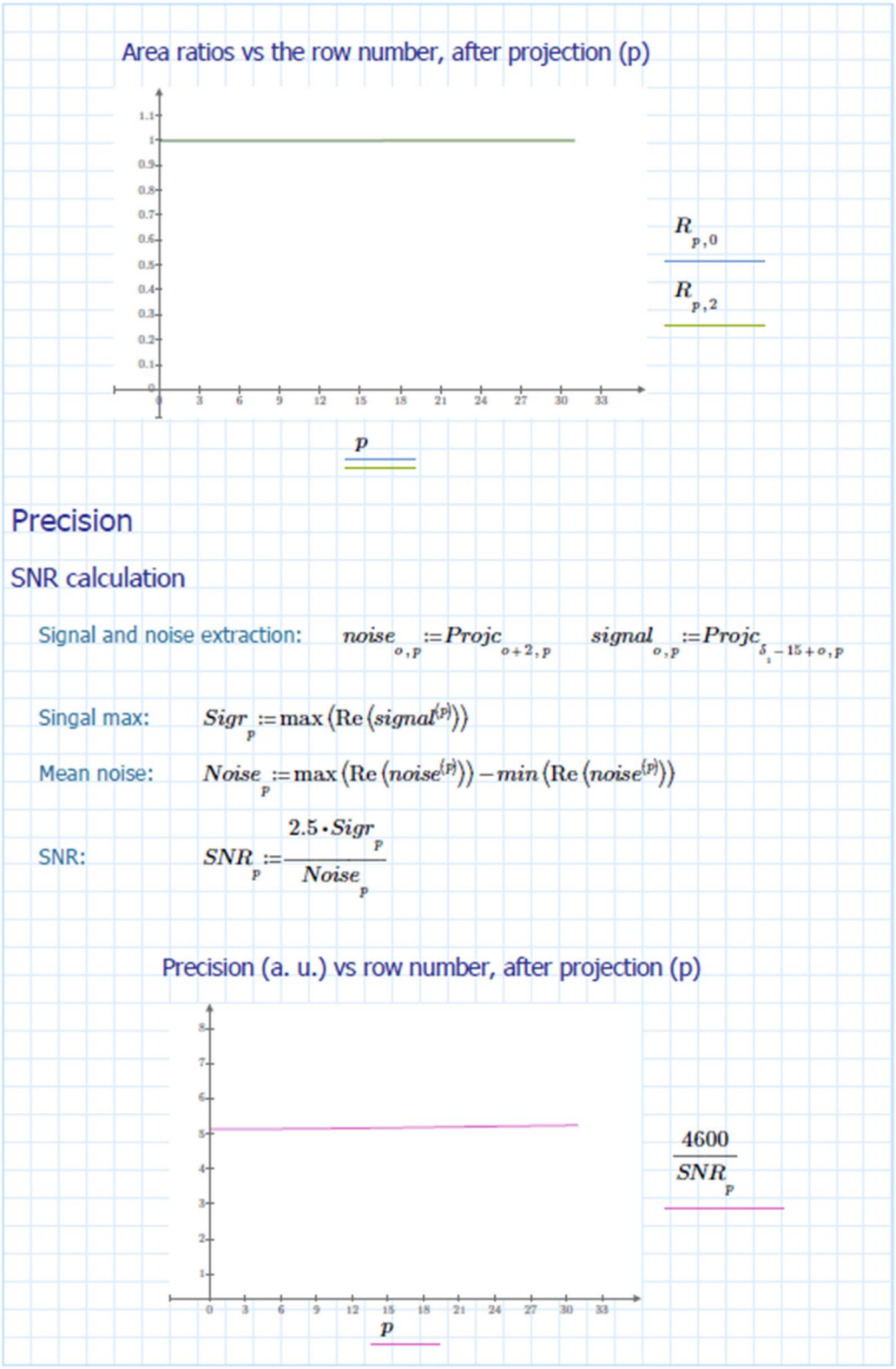


Figure S5: Screenshot of the PTC Mathcad Prime 9.0.0.0® file used for the simulations.

Section S3: DEFT optimization

Table S1: Trueness, represented by the highest difference with the reference (Δ_{ref}^{max}) and precision, represented by the highest relative standard deviation (RSD^{max}) on a reference Single Pulse experiment with different sampling time (AQ). For AQ < 1000 ms, linear prediction was used to reconstruct an FID of the same size as the 1000 ms one. All these experiments were obtained with the same time between scans.

AQ (ms)	1 000	750	500	250	125	75
Δ_{ref}^{max} (%)	Ref	0.37	0.39	0.24	0.47	0.65
RSD^{max} (%)	0.31	0.41	0.36	0.41	0.63	0.49

Section S4: R^2D^3 optimization on vanillin, vinyl acetate and cholesteryl acetate

Table S2: Highest difference with the reference single pulse (Δ_{ref}^{max}), RSD (RSD^{max}) and smallest signal to noise ratio (SNR^{min}) according to the number of added rows in the τ_r dimension and the apodization in the τ_r dimension, for a τ_r^{max} of 40 s. The values are highlighted based on whether they are below 0.5% (blue), between 0.5% and 1% (green) or between 1% and 5% (orange). Δ_{ref}^{max}, RSD^{max} and SNR^{min} were calculated on a series of 5 vinyl acetate (with $Cr(acac)_3$) R^2D^3 spectra.

	Nb rows	1	5	9	15	19	25	31	35	45	49	64
No apodization (τ_r dim.)	**Δ_{ref}^{max} (%)**	3.35	3.04	2.38	1.33	1.26	1.65	1.45	1.20	0.77	0.68	0.51
	RSD^{max} (%)	0.33	0.35	0.44	0.70	0.83	0.87	0.87	0.87	0.93	0.98	0.95
	SNR^{min}	442	457	450	368	347	332	302	289	274	263	253
Exponential apodization (τ_r dim.) (b = 2)	**Δ_{ref}^{max} (%)**	1.20	1.15	1.03	0.86	0.75	0.73	0.67	0.61	0.44	0.43	0.26
	RSD^{max} (%)	0.67	0.68	0.70	0.72	0.74	0.75	0.75	0.75	0.77	0.78	0.83
	SNR^{min}	393	388	377	356	342	323	306	304	278	272	253
Exponential apodization (τ_r dim.) (b = 8)	**Δ_{ref}^{max} (%)**	0.64	0.63	0.61	0.46	0.43	0.47	0.46	0.43	0.36	0.32	0.26
	RSD^{max} (%)	0.72	0.72	0.72	0.80	0.81	0.73	0.80	0.81	0.80	0.77	0.78
	SNR^{min}	321	320	317	310	305	296	288	282	270	266	253

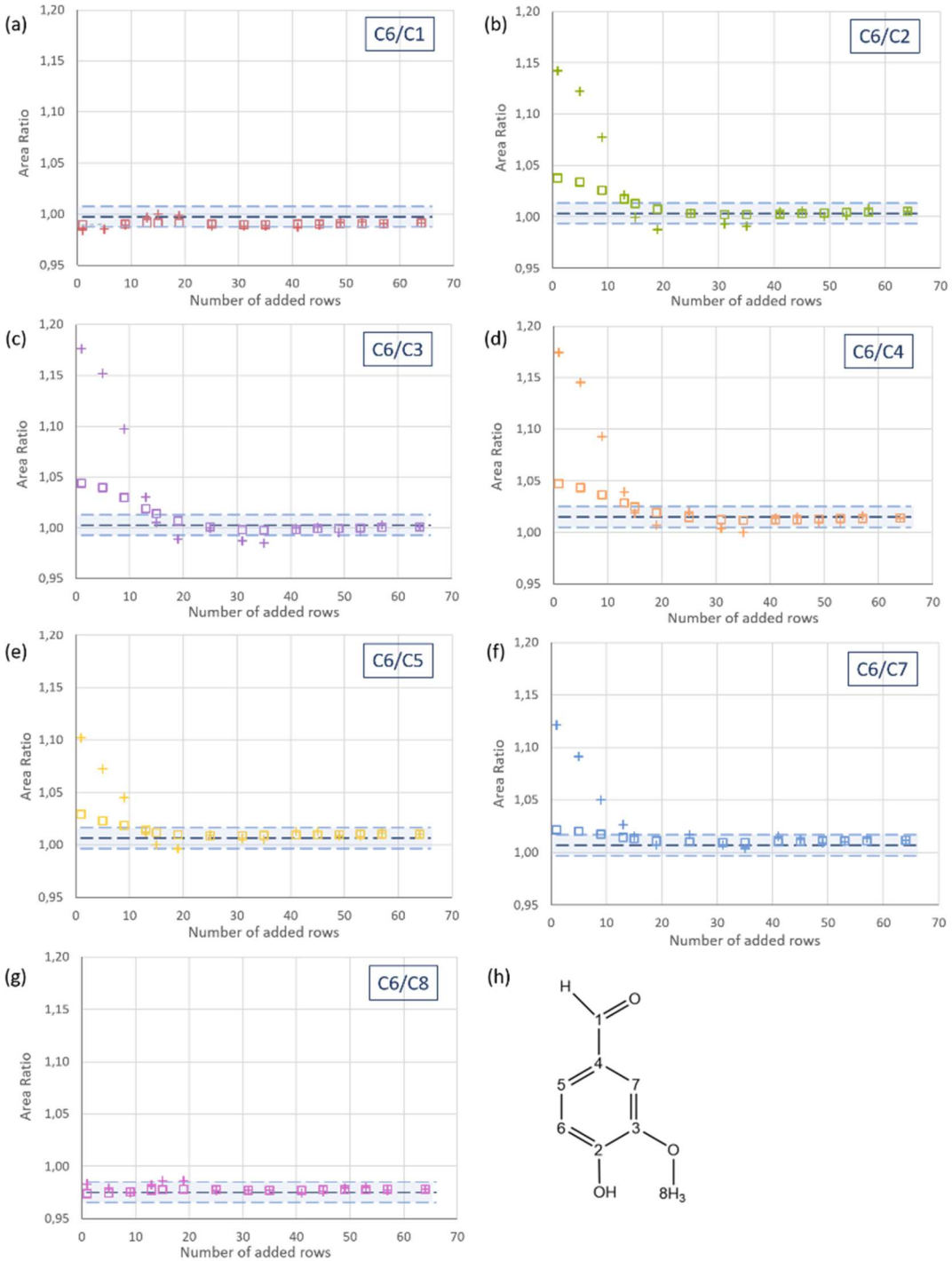


Figure S6: Area ratios C6/C1 (a), C6/C2 (b), C6/C3 (c), C6/C4 (d), C6/C5 (e), C6/C7 (f) and C8/C8 (g) ^{13}C positions of vanillin, depending on the number of rows that are added during the projection in the τ_r dimension. ^{13}C positions are reported in (h). On each graph, the area ratios without any apodization (+) and with an exponential one (b = 4) in the τ_r dimension (□) are represented. The dark dashed line is for the area ratio of the reference and the light dashed lines represents a ±1% difference with the reference. The T_1 are 1.3, 2.0, 2.3, 2.4, 1.1, 1.1, 1.2 and 1.2 for C1, C2, C3, C4, C5, C6, C7 and C8, respectively.

Table S3: Highest difference with the reference single pulse (Δ_{ref}^{max}), RSD (RSD^{max}) and smallest signal to noise ratio (SNR^{min}) according to the number of added rows in the τ_r dimension and the τ_r^{max}. All of the spectra were processed with an exponential apodization in the τ_r dimension (b = 4). The values are highlighted based on whether they are below 0.5% (blue), between 0.5% and 1% (green) or between 1% and 5% (orange). Δ_{ref}^{max}, RSD^{max} and SNR^{min} were calculated on a series of 5 vinyl acetate (with $Cr(acac)_3$) R^2D^3 spectra.

τ_r^{max} (s)	Nb rows	1	5	9	15	19	25	31	35	45	49	64
40	Δ_{ref}^{max} (%)	1.20	1.15	1.03	0.86	0.75	0.73	0.67	0.61	0.44	0.43	0.26
	RSD^{max} (%)	0.67	0.68	0.70	0.72	0.74	0.75	0.75	0.75	0.77	0.78	0.83
	SNR^{min}	393	388	377	356	342	323	306	304	278	272	253
20	Δ_{ref}^{max} (%)	1.43	1.37	1.24	1.01	0.96	0.96	0.82	0.89	0.73	0.67	0.44
	RSD^{max} (%)	0.68	0.69	0.73	0.75	0.77	0.79	0.81	0.82	0.84	0.84	0.83
	SNR^{min}	392	387	376	354	339	320	303	294	273	268	245
10	Δ_{ref}^{max} (%)	2.59	2.54	2.44	2.27	2.18	2.07	1.97	1.92	1.81	1.78	1.70
	RSD^{max} (%)	0.66	0.66	0.66	0.65	0.64	0.64	0.64	0.64	0.65	0.65	0.68
	SNR^{min}	378	373	365	347	336	319	305	297	280	274	253
5	Δ_{ref}^{max} (%)	3.29	3.25	3.16	3.04	3.00	2.98	2.98	3.06	3.25	3.30	3.44
	RSD^{max} (%)	0.66	0.68	0.71	0.76	0.79	0.80	0.79	0.79	0.78	0.79	0.87
	SNR^{min}	340	335	319	311	301	288	277	266	254	248	230
4	Δ_{ref}^{max} (%)	3.57	3.53	3.45	3.33	3.29	3.27	3.26	3.24	3.20	3.17	3.11
	RSD^{max} (%)	0.84	0.84	0.85	0.86	0.87	0.87	0.87	0.88	0.88	0.88	0.92
	SNR^{min}	346	341	331	312	300	284	271	263	247	241	224
3	Δ_{ref}^{max} (%)	2.74	2.74	2.74	2.82	2.92	3.11	3.31	3.41	3.65	3.72	3.94
	RSD^{max} (%)	0.47	0.47	0.50	0.60	0.68	0.80	0.90	0.93	1.05	1.08	1.20
	SNR^{min}	290	286	278	262	252	240	235	223	210	205	190
2	Δ_{ref}^{max} (%)	4.38	4.35	4.30	4.25	4.25	4.27	4.27	4.24	4.19	4.16	4.02
	RSD^{max} (%)	0.70	0.64	0.73	0.68	0.78	0.84	0.87	0.88	0.98	0.98	0.99
	SNR^{min}	330	326	317	300	289	276	265	258	244	240	223

Table S4: Chemical shifts and T_1 for every ^{13}C positions of cholesteryl acetate. The positions used in the article are highlighted in grey.

Site	Chem. Shift (ppm)	T_1 (s)
C1	170.17	3.0
C2	139.60	1.7
C3	122.59	0.5
C4	73.87	0.6
C5	56.72	0.6
C6	56.23	0.6
C7	50.08	0.6
C8	42.32	4.1
C9	39.79	0.3
C10	39.56	0.7
C11	38.16	0.3
C12	37.05	0.3
C13	36.58	4.7
C14	36.25	0.3
C15	35.84	0.6
C16	31.91	0.3
C17	31.89	0.5
C18	28.26	0.3
C19	28.00	1.8
C20	27.80	0.3
C21	24.31	0.3
C22	23.92	0.5
C23	22.84	1.4
C24	22.60	1.6
C25	21.32	1.1
C26	21.08	0.3
C27	19.30	1.0
C28	18.76	0.8
C29	11.88	1.1

Table S5: Difference with the reference single pulse (Δ_{ref}) and RSD for three sets of τ_r values, three data processing and two are ratios (C19/C13 for which the T_1 ratio is 1.8/4.7=1/2.6 and C26/C13 for which the T_1 ratio is 0.3/4.7=1/15.7). Δ_{ref} and RSD were calculated on a series of 5 cholesteryl acetate R^2D^3 spectra.

	τ_r param.	τ_r^{max}= 46,5; b=2; τ_r^{min}=0,5		τ_r^{max}= 4,2; b=2; τ_r^{min}=0,5		τ_r^{max}= 41; b=2; τ_r^{min}=6	
	Area ratio	C19/C13	C26/C13	C19/C13	C26/C13	C19/C13	C26/C13
1 row, no apodization (τ_r dim.)	Δ_{ref} (%)	52.17	62.61	65.44	83.74	6.59	2.48
	RSD (%)	1.20	1.63	1.50	1.83	1.90	1.52
64 rows, no apodization (τ_r dim.)	Δ_{ref} (%)	1.76	0.78	12.17	8.80	2.54	2.08
	RSD (%)	3.55	3.60	3.46	4.99	4.57	3.50
1 row corr., no apodization (τ_r dim.)	Δ_{ref} (%)	1.75	0.77	11.08	9.47	2.57	2.09
	RSD (%)	1.20	1.63	1.87	1.89	1.90	1.52

Table S6: Highest difference with the reference single pulse (Δ_{ref}^{max}) and RSD (RSD^{max}) according to the number of added rows in the τ_r dimension for different τ_r functions and apodization. The values are highlighted based on whether they are below 0.5% (blue). between 0.5% and 1% (green) or between 1% and 5% (orange). Δ_{ref}^{max} and RSD^{max} were calculated on a series of 5 vanillin R^2D^3 spectra.

τ_r^{max} (s)	Process	Nb rows	1	5	9	13	15	19	25	31	35	41	45	49	53	57	64
26.1	No apodization (τ_r dim.)	Δ_{ref}^{max} (%)	17.29	14.86	9.45	2.75	1.13	1.55	0.98	1.51	1.74	0.98	0.84	0.71	0.62	0.67	0.63
		RSD^{max} (%)	0.41	0.34	0.27	0.34	0.35	0.35	0.41	0.43	0.47	0.53	0.54	0.61	0.65	0.68	0.73
	Exponential apodization (τ_r dim.) (LB 4k)	Δ_{ref}^{max} (%)	4.14	3.66	2.71	1.63	1.15	0.63	0.70	0.75	0.76	0.74	0.71	0.67	0.65	0.64	0.63
		RSD^{max} (%)	0.29	0.29	0.32	0.31	0.33	0.33	0.41	0.46	0.50	0.56	0.59	0.62	0.65	0.68	0.71
13.1	No apodization (τ_r dim.)	Δ_{ref}^{max} (%)	21.85	19.30	13.59	6.48	3.74	1.65	2.02	-0.57	-0.98	-0.98	-0.94	-0.84	-0.86	-0.79	-0.70
		RSD^{max} (%)	0.48	0.44	0.29	0.25	0.27	0.28	0.26	0.38	0.44	0.53	0.53	0.48	0.47	0.51	0.56
6.5	//	Δ_{ref}^{max} (%)	27.73	25.27	19.57	12.25	9.24	6.88	6.74	4.15	3.09	3.56	3.12	2.12	1.84	2.26	1.76
		RSD^{max} (%)	0.38	0.66	0.43	0.41	0.45	0.41	0.34	0.48	0.59	0.65	0.67	0.69	0.70	0.70	0.70
3.3	//	Δ_{ref}^{max} (%)	34.81	32.77	27.88	21.22	18.35	15.78	15.35	12.70	11.61	11.94	10.50	10.48	10.13	10.42	9.72
		RSD^{max} (%)	0.50	0.51	0.51	0.47	0.49	0.56	0.67	0.70	0.69	0.61	2.31	0.59	0.61	0.61	0.65
1.7	//	Δ_{ref}^{max} (%)	42.90	41.46	37.59	32.03	29.56	27.26	26.83	24.40	23.35	23.58	23.12	22.21	21.91	22.23	21.77
		RSD^{max} (%)	0.54	0.57	0.49	0.73	0.82	0.88	0.82	0.82	0.82	0.89	0.96	1.00	0.99	0.92	0.93
1.7 (b: 2)	//	Δ_{ref}^{max} (%)	46.64	45.37	41.84	36.49	33.98	31.32	30.14	27.06	25.67	25.46	24.71	23.55	23.06	23.19	22.21
		RSD^{max} (%)	0.48	0.46	0.45	0.52	0.65	0.73	0.72	0.84	0.85	0.79	0.82	0.84	0.82	0.79	0.80
1.7 (b: 0.5)	//	Δ_{ref}^{max} (%)	37.97	36.37	32.71	27.73	25.71	24.39	25.01	23.20	22.44	22.94	22.60	21.78	21.56	21.93	21.44
		RSD^{max} (%)	0.59	0.55	0.55	0.70	0.82	0.96	0.98	0.98	0.95	0.87	0.79	0.80	1.31	0.71	0.80

Table S7: Vinyl acetate experimental time and time saving factor, compared to the reference single pulse with $\tau_r = 10 \cdot T_1^{max}$ for different τ_r^{max}.

τ_r^{max} (s)	Exp time	Time saving factor
40	3 min 08 s	4.4
20	1 min 42 s	8.0
10	59 s	13.9
5	37 s	22.2
4	33 s	24.8
3	29 s	28.0
2	24 s	34.2

Table S8: Vanillin experimental time and time saving factor, compared to the reference single pulse with $\tau_r = 10 \cdot T_1^{max}$ for different τ_r^{max}.

τ_r^{max} (s)	Exp time	Time saving factor
26.1	47 min 17 s	4.3
13.1	26 min 05 s	7.8
6.5	16 min 21 s	12.4
3.3	11 min 37 s	17.5
1.7	9 min 16 s	21.9
1.7 (b:2)	8 min 47 s	23.2
1.7 (b:0.5)	10 min 17 s	19.8

Table S9: Cholesteryl acetate (1M in $CDCl_3$) experimental time and time saving factor for three sets of τ_r values, compared to the reference single pulse with $\tau_r = 10 \cdot T_1^{max}$ of 1 hour 58 min 31 s.

$\tau_{r\,max}$ (s)	Exp time	Time saving factor
47 (τ_r^{min}: 0.5)	18 min 32 s	6.4
5 (τ_r^{min}: 0.5)	3 min 52 s	30.6
47 (τ_r^{min}: 6)	29 min 50 s	4.0

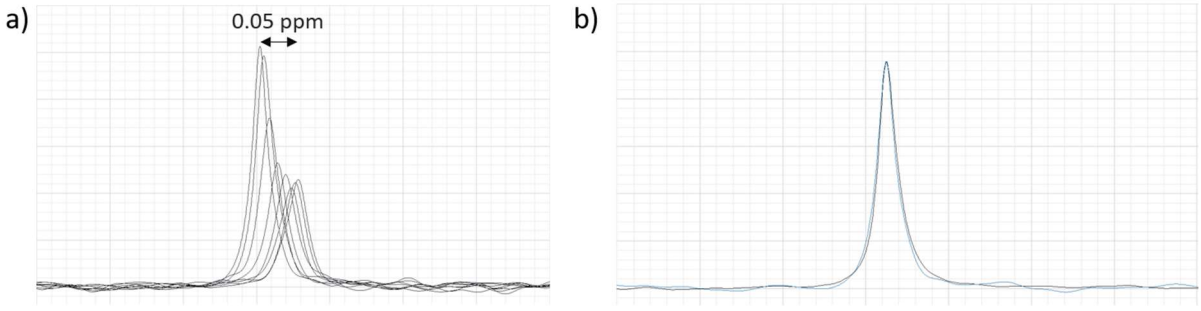


Figure S7: Cholesteryl acetate spectra from 170.5 to 169.8 ppm (C1). (a) R^2D^3 rows before processing in the τ_r dimension and with LB = 0.3 Hz in the direct dimension; and (b) R^2D^3 spectrum after processing in the τ_r dimension and with LB = 2 Hz in the direct dimension (blue), and single pulse spectrum with LB = 2 Hz (black). The τ_r function was: τ_r^{max} = 46.5 s. b = 2. τ_r^{min} = 0.5 s.

Table S10: Maximum deviation between two τ_r values (D^{max}) measured for each carbon of cholesteryl acetate and for 3 sets of τ_r.

τ_r^{max}: 41; b: 2; τ_r^{min}: 6	
Site	D^{max} (ppm)
C1	0.009
C2	0.009
C3	0.002
C4	0.003
C5	0.008
C6	0.01
C7	0.009
C8	0.01
C9	0.007
C10	0.01
C11	0.004
C12	0.007
C13	0.006
C14	0.006
C15	0.0006
C17	0.01
C18	0
C19	0.004
C20	0.008
C21	0.004
C22	0.002
C23	0.004
C24	0.002
C25	0.006
C26	0.006
C27	0.0003
C28	0.003
C29	0.004
min:	0.000
max:	0.010
average:	0.005

τ_r^{max}: 4.2; b: 2; τ_r^{min}: 0.5	
Site	D^{max} (ppm)
C1	0.038
C2	0.035
C3	0.006
C4	0.009
C5	0.025
C6	0.036
C7	0.037
C8	0.025
C9	0.027
C10	0.013
C11	0.02
C12	0.022
C13	0.02
C14	0.019
C15	0.0006
C17	0.024
C18	0.004
C19	0
C20	0.019
C21	0.009
C22	0.01
C23	0.01
C24	0.004
C25	0.02
C26	0.017
C27	0.002
C28	0.013
C29	0.009
min:	0.000
max:	0.038
average:	0.017

τ_r^{max}: 46.5; b: 2; τ_r^{min}: 0.5	
Site	D^{max} (ppm)
C1	0.053
C2	0.048
C3	0.01
C4	0.013
C5	0.036
C6	0.05
C7	0.052
C8	0.034
C9	0.038
C10	0.018
C11	0.028
C12	0.029
C13	0.029
C14	0.024
C15	0.003
C17	0.038
C18	0.003
C19	0.002
C20	0.027
C21	0.012
C22	0.016
C23	0.014
C24	0.004
C25	0.028
C26	0.024
C27	0.002
C28	0.019
C29	0.014
min:	0.002
max:	0.053
average:	0.024